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**PROVISIONAL APPLICATION FOR PATENT COVER SHEET**

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

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INVENTOR(S)			
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		Residence (City and either State or Foreign Country)	
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Additional inventors are being named on the <u>1</u> separately numbered sheets attached hereto			
TITLE OF THE INVENTION (500 characters max)			
Aqueous Emulsions using Silicone Polyether Block Copolymers			
Direct all correspondence to: CORRESPONDENCE ADDRESS			
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ENCLOSED APPLICATION PARTS (check all that apply)			
<input checked="" type="checkbox"/> Specification Number of Pages <u>28</u>		<input type="checkbox"/> CD(s), Number _____	
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<input type="checkbox"/> Application Data Sheet. See 37 CFR 1.76			
METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT			
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27.		FILING FEE Amount (\$)	
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[Page 1 of 2]

Respectfully submitted,

SIGNATURE

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989-496-3101

Date

09/17/2004

REGISTRATION NO.

45,260

(If appropriate)

Docket Number:

DC10104PSP1

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Docket Number **DC10104PSP1**

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[Page 2 of 2]

Number 2 of 2

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<b>CERTIFICATE OF MAILING BY "EXPRESS MAIL" (37 CFR 1.10)</b>			Docket No.	
Applicant(s): Shaow Burn Lin			DC10104 PSP 1	
Application No.	Filing Date	Examiner	Customer No.	Group Art Unit
			00137	

Invention: Aqueous Emulsions using Silicone Polyether Block Copolymers

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**UNITED STATES PATENT APPLICATION  
FOR**

**Aqueous Emulsions**

**using**

**Silicone Polyether Block Copolymers**

**Having the inventor(s):**

**Shaow Burn Lin**

**Kimmai Nguyen**

**FIELD OF THE INVENTION**

This application relates to emulsion compositions from  $(AB)_n$  silicone polyether block copolymers, methods for preparing the emulsion compositions, and personal, household, and healthcare formulations containing the emulsion compositions.

## BACKGROUND OF THE INVENTION

Long-standing needs in the field of cosmetic and drug formulation/delivery field are to identify aqueous based dispersion compositions that entrap actives easily, are stable under various chemical and mechanical stresses, and yet are able to deliver the actives in a controlled manner under desired conditions. Aqueous dispersions or emulsions derived from silicone surfactants, and more particularly silicone polyether surfactants, are of interest because of additional inherent benefits that this class of surfactants possesses vs. other types. For example, silicone polyether surfactants often have improved aesthetics in personal care formulations.

The present inventors have discovered that certain  $(AB)_n$  silicone polyethers form stable dispersions in aqueous media and can be used to create emulsions. These stable dispersions can be used to formulate compositions for the delivery of pharmaceutical and personal care actives.

While  $(AB)_n$  silicone polyether block copolymers are known, the selection of the specific structures or certain molecular variables that enables the copolymers to form stable dispersions in aqueous media is heretofore unknown.

U.S. Patents Nos. 3,957,842 and 4,022,941 by Prokai et al. teach linear siloxane-polyoxyalkylene  $(AB)_n$  block copolymers having an average molecular weight of at least 30,000. The '842 and '941 patents further teach the use of the linear siloxane-polyoxyalkylene  $(AB)_n$  block copolymers in polyurethane forming compositions, as well as the cured foams and articles produced therefrom.

U.S. Patent Nos. 5,472,686 and 5,660,819 by Tsubaki et al. teach cosmetic formulations containing a linear polysiloxane-polyoxyalkylene block copolymer as the main component. However, the '686 and '819 patents do not teach polysiloxane-polyoxyalkylene block copolymers structures that stabilize emulsions.

U.S. Patent 5,767,219 teaches a polysiloxane-polyether block copolymer having excellent heat resistance, which is characterized by the molecular structure consisting of repetition of diorganopolysiloxane units and polyoxyalkylene units having a bisphenol

linkage of the formula  $-O-Pn-CMe_2-Pn-O-$  in the unit, Pn being a 1,4-phenylene group and Me is a methyl group.

5 The present inventors have discovered that a particular class of silicone polyethers, namely  $(AB)_n$  block copolymer silicone polyethers, form emulsion compositions in aqueous media. The emulsions possess enhanced stability and are useful to prepare formulations for the delivery of personal, household, and healthcare active materials.



## SUMMARY OF THE INVENTION

5       The present invention relates to a water continuous emulsion composition comprising  
a  $(AB)_n$  silicone polyether copolymer wherein the emulsion has an average particle size of  
less 10 micrometers. Preferably, the emulsion composition comprises a  $(AB)_n$  silicone  
polyether copolymer that is a polyorganosiloxane-polyoxyalkylene block copolymer having  
the average formula;



10       where

x and y are greater than 4, m is from 2 to 4 inclusive, z is greater than 2,

R is independently a monovalent organic group,

$R^1$  is a divalent hydrocarbon containing 2 to 30 carbons.

15       The invention further provides a process for preparing a water continuous emulsion  
having an average particle size of less than 10 micrometers comprising;

I) mixing

A)  $(AB)_n$  silicone polyether copolymer

B) a water miscible volatile solvent

20       to form a hydrophobic phase,

II) adding water to the hydrophobic phase to form the water continuous emulsion.

Furthermore, the present invention relates to personal, household, and healthcare  
formulations containing the emulsion compositions.

25

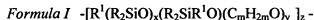
## DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a water continuous emulsion composition comprising  
 5 a (AB)<sub>n</sub> silicone polyether copolymer wherein the emulsion has an average particle size of less than 10 micrometers. The emulsion compositions of the present invention comprise a hydrophobic phase dispersed as discrete particles in a water continuous phase. The hydrophobic phase particles are stabilized in the emulsion by presence of a (AB)<sub>n</sub> silicone polyether copolymer, as discussed infra. The dispersed hydrophobic phase particles have an  
 10 average particle size that is less than 10 micrometers, alternatively less than 5 micrometers, or alternatively less than 1 micrometers. "Average particle size" is the accepted meaning in the emulsion art, and can be determined for example using a particle size analyzer such as a *Nanotrac 150*.

The water continuous emulsions of the present invention can be furthermore  
 15 considered as stable water continuous emulsions. For purposes of this invention, "stable water continuous emulsion" means that the emulsion's average particle size distribution does not change substantially within a given period of time. Stability can be evaluated by storing samples of the emulsion at room temperature for a period of time and observing the emulsions for any signs of instability, such as separation or creaming. Samples can also be  
 20 stored at elevated temperatures, for example 40°C, to simulate an accelerate aging process. Typically, the emulsions of the present invention do not show separation at room temperature after aging for at least 4 weeks. Typically the average particle size remains less than 10 μm particle size for at least 4 weeks at room temperature.

The emulsion compositions of the present invention comprise a (AB)<sub>n</sub> silicone  
 25 polyether copolymer, which stabilizes the emulsion. The (AB)<sub>n</sub> silicone polyether copolymer can be selected from any (AB)<sub>n</sub> silicone polyether copolymer structure that has sufficient surfactant or amphiphilic character. Preferably, the (AB)<sub>n</sub> silicone polyether copolymer is a polyorganosiloxane-polyoxyalkylene block copolymer having the average formula;

30



where

x and y are greater than 4, m is from 2 to 4 inclusive, z is greater than 2,

R is independently a monovalent organic group,

R<sup>1</sup> is a divalent hydrocarbon containing 2 to 30 carbons.

5

The siloxane block in Formula I is a predominately linear siloxane polymer having the formula (R<sub>2</sub>SiO)<sub>x</sub>, wherein R is independently selected from a monovalent organic group, x is an integer greater than 4, alternatively x ranges from 5 to 19, or from 5 to 15.

10 The organic groups represented by R in the siloxane polymer are free of aliphatic unsaturation. These organic groups may be independently selected from monovalent hydrocarbon and monovalent halogenated hydrocarbon groups free of aliphatic unsaturation. These monovalent groups may have from 1 to 20 carbon atoms, alternatively 1 to 10 carbon atoms, and are exemplified by, but not limited to alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, undecyl, and octadecyl; cycloalkyl such as cyclohexyl; aryl  
15 such as phenyl, tolyl, xylyl, benzyl, and 2-phenylethyl; and halogenated hydrocarbon groups such as 3,3,3-trifluoropropyl, 3-chloropropyl, and dichlorophenyl. At least 50 percent, alternatively at least 80%, of the organic groups in the organopolysiloxane may be methyl (denoted as Me). Typically, the siloxane block is a predominately linear polydimethylsiloxane having the formula (Me<sub>2</sub>SiO)<sub>x</sub>, where x is as defined above.

20 The polyoxyalkylene block of the silicone polyether is represented by the formula (C<sub>m</sub>H<sub>2m</sub>O)<sub>y</sub>, wherein m is from 2 to 4 inclusive, and y is greater than 4, alternatively y can range from 5 to 30, or alternatively from 5 to 22. The polyoxyalkylene block typically can comprise oxyethylene units (C<sub>2</sub>H<sub>4</sub>O)<sub>y</sub>, oxypropylene units (C<sub>3</sub>H<sub>6</sub>O)<sub>y</sub>, oxybutylene units (C<sub>4</sub>H<sub>8</sub>O)<sub>y</sub>, or mixtures thereof. Typically, the polyoxyalkylene block comprises oxyethylene  
25 units (C<sub>2</sub>H<sub>4</sub>O)<sub>y</sub>.

At least one end of each polyoxyalkylene block in Formula I is linked to a siloxane block by a divalent organic group, designated R<sup>1</sup>. This linkage is determined by the reaction employed to prepare the (AB)<sub>n</sub> block silicone polyether copolymer. The divalent organic groups of R<sup>1</sup> may be independently selected from divalent hydrocarbons containing 2 to 30  
30 carbons and divalent organofunctional hydrocarbons containing 2 to 30 carbons. Representative, non-limiting examples of such divalent hydrocarbon groups include;

ethylene, propylene, butylene, pentylene, hexylene, heptylene, octylene, and the like. Representative, non-limiting examples of such divalent organofunctional hydrocarbons groups include acrylate and methacrylate. Typically,  $R^1$  is propylene,  $(-CH_2CH_2CH_2-)$ .

- The  $(AB)_n$  block silicone polyethers are endblocked. The endblocking unit is also
- 5 determined by the reaction employed to prepare the  $(AB)_n$  block silicone polyether copolymer, which is generally the residual reactive groups of the reactants used. For example, the  $(AB)_n$  block silicone polyether copolymers can be prepared by the metal catalyzed hydrosilylation reaction of a diallyl polyether (i.e. an allyl group is present on each molecular terminal end) with a SiH terminated polyorganosiloxane. The resulting  $(AB)_n$
- 10 block silicone polyether copolymer would have polyoxyalkylene blocks linked to the silicone blocks via a propyleneoxy group  $(-CH_2CH_2CH_2O-)$ , and using a slight molar excess of the allyl polyether would result in an allyl endblock unit  $(-CH_2CHCH_2)$ . Alternative endblock units can result from the addition of other molecules in the reaction employed to prepare the  $(AB)_n$  block silicone polyether copolymer that are capable of reacting with the siloxane or
- 15 polyether block intermediates. For example, the addition of organic compounds having mono-terminated aliphatic unsaturation (such as a mono allyl terminated polyether) will result in the endcapping of the  $(AB)_n$  block silicone polyether copolymer with that organic compound. Preferably, the endblocking units of the  $(AB)_n$  block silicone polyether is an allyl ether  $(CH_2=CHCH_2O-)$  or allyl polyether.
- 20 The molecular weights of the  $(AB)_n$  block silicone polyether copolymers will be determined by the number of repeating siloxane and polyoxyethylene blocks, as indicated by the subscript  $z$  in Formula 1. Typically, the value of  $z$  is such to provide weight average molecular weights ( $M_w$ ) to range from 1,500 to 150,000, alternatively, from 10,000 to 100,000.
- 25 The  $(AB)_n$  SPEs useful to prepare the emulsion compositions of the present invention can be prepared by any method known in the art for preparing such block copolymers. Typically however, the  $(AB)_n$  SPEs useful in the preparation of the compositions of the present invention are obtained from a method comprising reacting an SiH terminated organopolysiloxane with a polyoxyethylene having an unsaturated hydrocarbon group at each
- 30 molecular terminal, in a hydrosilylation reaction, wherein the mole ratio of the unsaturated hydrocarbon groups to SiH in the reaction is at least 1:1.

The emulsion compositions of the present invention can be prepared by mixing the  $(AB)_n$  SPEs with water using any technique known in the state of the art for creating emulsion compositions. The type and extent of the mixing technique will depend on the specific structure of the  $(AB)_n$  SPE chosen. For example, some  $(AB)_n$  SPEs will form emulsion compositions spontaneously when mixed with water, while others  $(AB)_n$  SPEs will require the presence of a water soluble solvent to facilitate the formation of the emulsion. In a preferred embodiment, the emulsion compositions of the present invention are prepared using a process comprising:

I) mixing

A)  $(AB)_n$  silicone polyether copolymer

B) a water miscible volatile solvent

to form a hydrophobic phase,

II) adding water to the hydrophobic phase to form the water continuous emulsion.

The  $(AB)_n$  SPEs, component A) in step I), is the same as described above. Component B) is a water-miscible volatile solvent. As used herein "water-miscible" means the solvent forms a dispersion with water at room temperature for at least several hours. "Volatile" means the solvent has a higher vapor pressure than water at various temperatures. As such, when the aqueous dispersion of the organopolysiloxane and solvent are subjected to conditions to remove the solvent, such as heating the dispersion under reduced pressures, the solvent is primarily removed first, allowing all or most of the water to remain in the composition.

Suitable water-miscible volatile solvents for vesicle dispersion preparation include organic solvents such as alcohols, ethers, glycols, esters, acids, halogenated hydrocarbons, diols. The organic solvents should be miscible with water at the proportion and lower in order to effectively disperse silicones and maintain stable and uniform dispersion overtime. For the purpose of illustration, water-miscible alcohols include methanol, ethanol, propanol, isopropanol, butanol, and higher hydrocarbon alcohols; ethers include glycol ethers, methyl-ethyl ether, methyl isobutyl ether (MIBK), etc; glycols include propylene glycols, esters include esters of triglycerol, the esterification products of acid and alcohol; halogenated hydrocarbons include chloroform. Typically water-miscible organic solvents are solvents

- with relatively low boiling points ( $<100^{\circ}\text{C}$ ) or high evaporation rate, so they may be removed under vacuum with ease. The most preferred water-miscible organic solvents for this invention are volatile alcohols including methanol, ethanol, isopropanol, and propanol. These alcohols can be removed from aqueous mixtures containing silicone vesicle dispersions via vacuum stripping at ambient temperature.

After forming a hydrophobic phase of A) and B), water is then added to the mixture. There are no special requirements or conditions needed for effecting the mixing of components A), B) in step I and subsequent mixing with water in step II). The mixing and water addition steps can be conducted in a batch, semi-continuous, or continuous process.

- The hydrophobic phase of step I) can also comprise component C) a silicone or organic oil. The silicone can be any organopolysiloxane having the general formula  $\text{R}_i\text{SiO}_{(4-i)/2}$  in which  $i$  has an average value of one to three and  $\text{R}$  is a monovalent organic group. The organopolysiloxane can be cyclic, linear, branched, and mixtures thereof.
- In one embodiment, the silicone selected as component C) is a volatile methyl siloxane (VMS) which includes low molecular weight linear and cyclic volatile methyl siloxanes. Volatile methyl siloxanes conforming to the CTFA definition of cyclomethicones are considered to be within the definition of low molecular weight siloxane.

- Linear VMS have the formula  $(\text{CH}_3)_3\text{SiO}\{(\text{CH}_3)_2\text{SiO}\}_f\text{Si}(\text{CH}_3)_3$ . The value of  $f$  is 0-7. Cyclic VMS have the formula  $\{(\text{CH}_3)_2\text{SiO}\}_g$ . The value of  $g$  is 3-6. Preferably, these volatile methyl siloxanes have a molecular weight of less than 1,000; a boiling point less than  $250^{\circ}\text{C}$ ; and a viscosity of 0.65 to 5.0 centistoke ( $\text{mm}^2/\text{s}$ ), generally not greater than 5.0 centistoke ( $\text{mm}^2/\text{s}$ ).

- Representative linear volatile methyl siloxanes are hexamethyldisiloxane (MM) with a boiling point of  $100^{\circ}\text{C}$ , viscosity of  $0.65 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiOSiMe}_3$ ;
- octamethyltrisiloxane (MDM) with a boiling point of  $152^{\circ}\text{C}$ , viscosity of  $1.04 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiOMe}_2\text{SiOSiMe}_3$ ; decamethyltetrasiloxane ( $\text{MD}_2\text{M}$ ) with a boiling point of  $194^{\circ}\text{C}$ , viscosity of  $1.53 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_2\text{SiMe}_3$ ;
- dodecamethylpentasiloxane ( $\text{MD}_3\text{M}$ ) with a boiling point of  $229^{\circ}\text{C}$ , viscosity of  $2.06 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_3\text{SiMe}_3$ ; tetradecamethylhexasiloxane ( $\text{MD}_4\text{M}$ ) with

a boiling point of 245 °C, viscosity of 2.63 mm<sup>2</sup>/s, and formula Me<sub>3</sub>SiO(Me<sub>2</sub>SiO)<sub>4</sub>SiMe<sub>3</sub>; and hexadecamethylheptasiloxane (MD<sub>5</sub>M) with a boiling point of 270 °C, viscosity of 3.24 mm<sup>2</sup>/s, and formula Me<sub>3</sub>SiO(Me<sub>2</sub>SiO)<sub>5</sub>SiMe<sub>3</sub>.

- Representative cyclic volatile methyl siloxanes are hexamethylcyclotrisiloxane (D<sub>3</sub>),
- 5 a solid with a boiling point of 134 °C, a molecular weight of 223, and formula {(Me<sub>2</sub>)SiO}<sub>3</sub>; octamethylcyclotetrasiloxane (D<sub>4</sub>) with a boiling point of 176 °C, viscosity of 2.3 mm<sup>2</sup>/s, a molecular weight of 297, and formula {(Me<sub>2</sub>)SiO}<sub>4</sub>; decamethylcyclopentasiloxane (D<sub>5</sub>) with a boiling point of 210 °C, viscosity of 3.87 mm<sup>2</sup>/s, a molecular weight of 371, and formula {(Me<sub>2</sub>)SiO}<sub>5</sub>; and dodecamethylcyclohexasiloxane (D<sub>6</sub>) with a boiling point of 245
- 10 °C, viscosity of 6.62 mm<sup>2</sup>/s, a molecular weight of 445, and formula {(Me<sub>2</sub>)SiO}<sub>6</sub>.

- The silicone selected as component C) can be any polydiorganosiloxane fluid, gum, or mixtures thereof. If the polyorganosiloxane has a molecular weight equal to or greater than 1000, it can be blended with the volatile methyl siloxanes described above. The polydiorganosiloxane gums suitable for the present invention are essentially composed of
- 15 dimethylsiloxane units with the other units being represented by monomethylsiloxane, trimethylsiloxane, methylvinylsiloxane, methylethylsiloxane, diethylsiloxane, methylphenylsiloxane, diphenylsiloxane, ethylphenylsiloxane, vinyl ethylsiloxane, phenylvinylsiloxane, 3,3,3-trifluoropropylmethylsiloxane, dimethylphenylsiloxane, methylphenylvinylsiloxane, dimethylethylsiloxane, 3,3,3-trifluoropropyldimethylsiloxane,
- 20 mono-3,3,3-trifluoropropylsiloxane, aminoalkylsiloxane, monophenylsiloxane, monovinylsiloxane and the like.

- When component C) is an organic oil, it may be selected from any organic oil known in the art suitable for use in the preparation of personal, household, or healthcare formulations. Suitable organic oils include, but are not limited to, natural oils such as
- 25 coconut oil; hydrocarbons such as mineral oil and hydrogenated polyisobutene; fatty alcohols such as octyldodecanol; esters such as C12 -C15 alkyl benzoate; diesters such as propylene dipelarganate; and triesters, such as glyceryl trioctanoate. The organic oil components can also be mixture of low viscosity and high viscosity oils. Suitable low viscosity oils have a viscosity of 5 to 100 mPa.s at 25°C, and are generally esters having the structure RCO-OR'

- wherein RCO represents the carboxylic acid radical and wherein OR' is an alcohol residue. Examples of these low viscosity oils include isotridecyl isononanoate, PEG-4 diheptanoate, isostearyl neopentanoate, tridecyl neopentanoate, cetyl octanoate, cetyl palmitate, cetyl ricinoleate, cetyl stearate, cetyl myristate, coco-dicaprylate/caprate, decyl isostearate,
- 5 isodecyl oleate, isodecyl neopentanoate, isohexyl neopentanoate, octyl palmitate, dioctyl malate, tridecyl octanoate, myristyl myristate, octododecanol, or mixtures of octyldodecanol, acetylated lanolin alcohol, cetyl acetate, isododecanol, polyglyceryl-3-diisostearate, or mixtures thereof. The high viscosity surface oils generally have a viscosity of 200-1,000,000 mPa.s at 25°C, preferably a viscosity of 100,000-250,000 mPa.s. Surface oils include castor
- 10 oil, lanolin and lanolin derivatives, triisocetyl citrate, sorbitan sesquioleate, C10-18 triglycerides, caprylic/capric triglycerides, coconut oil, corn oil, cottonseed oil, glyceryl triacetyl hydroxystearate, glyceryl triacetyl ricinoleate, glyceryl trioctanoate, hydrogenated castor oil, linseed oil, mink oil, olive oil, palm oil, illipe butter, rapeseed oil, soybean oil, sunflower seed oil, tallow, tricaprin, trihydroxystearin, triisostearin, trilaurin, trilinolein,
- 15 trimyristin, triolein, tripalmitin, tristearin, walnut oil, wheat germ oil, cholesterol, or mixtures thereof. Mention may be made, among the optional other non-silicone fatty substances, of mineral oils, such as liquid paraffin or liquid petroleum, of animal oils, such as perhydrosqualene or arara oil, or alternatively of vegetable oils, such as sweet almond, calophyllum, palm, castor, avocado, jojoba, olive or cereal germ oil. It is also possible to use
- 20 esters of lanolic acid, of oleic acid, of lauric acid, of stearic acid or of myristic acid, for example; alcohols, such as oleyl alcohol, linoleyl or linolenyl alcohol, isostearyl alcohol or octyldodecanol; or acetylglycerides, octanoates, decanoates or ricinoleates of alcohols or of polyalcohols. It is alternatively possible to use hydrogenated oils which are solid at 25°C, such as hydrogenated castor, palm or coconut oils, or hydrogenated tallow; mono-, di-, tri- or
- 25 sucroglycerides; lanolins; or fatty esters which are solid at 25°C.

- The hydrophobic phase of step I) can also comprise optionally component D) a personal, household, or healthcare active. A listing of possible personal, household, or health care ingredients is taught in WO 03/101412, which is incorporated herein by reference. The personal or health care ingredient can also be selected from a personal or health care "active",
- 30 that is, any compound known to have either cosmetic and/or pharmaceutical activity. A representative listing of such personal or health care actives are disclosed in US Patent



6,168,782, which is hereby incorporated by reference. The common assignee's U.S. Patent 5,948,855 (September 7, 1999), also contains an extensive list of some appropriate oil soluble active ingredients such as vitamins and drugs which can be used in the oil phase of the oil in water emulsions, among which are vitamins, including but not limited to, Vitamin A<sub>1</sub>,

- 5 RETINOL, C<sub>2</sub>-C<sub>18</sub> esters of RETINOL, Vitamin E, TOCOPHEROL, esters of Vitamin E, and mixtures thereof. RETINOL includes trans-RETINOL, 13-cis-RETINOL, 11-cis-RETINOL, 9-cis-RETINOL, and 3,4-didehydro-RETINOL. Other vitamins which are appropriate include RETINYL ACETATE, RETINYL PALMITATE, RETINYL PROPIONATE,  $\alpha$ -TOCOPHEROL, TOCOPHERSOLAN, TOCOPHERYL ACETATE, 10 TOCOPHERYL LINOLEATE, TOCOPHERYL NICOTINATE, and TOCOPHERYL SUCCINATE.

The amount of components A), B), C), and D) can vary in the process, but typically range as follows;

- 15 A) 2 to 60 wt%, alternatively 2 to 50 wt %, or alternatively 2 to 40 wt%,  
B) 0 to 50 wt%, alternatively 2 to 30 wt %, or alternatively 2 to 20 wt%,  
C) 0 to 30 wt%, alternatively 0 to 25 wt %, or alternatively 0 to 20 wt%,  
D) 0 to 30 wt%, alternatively 0 to 25 wt %, or alternatively 0 to 20 wt%,

where sufficient amount of water is added to provide the sum of the wt% of A), B), C), D), and water to equal 100%.

- 20 Compositions prepared according to the invention can be used in various over-the-counter (OTC) personal care compositions, health care compositions, and household care compositions, but especially in the personal care arena. Thus, they can be used in antiperspirants, deodorants, skin creams, skin care lotions, moisturizers, facial treatments such as acne or wrinkle removers, personal and facial cleansers, bath oils, perfumes, 25 colognes, sachets, sunscreens, pre-shave and after-shave lotions, liquid soaps, shaving soaps, shaving lathers, hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories, hair cuticle coats, make-ups, color cosmetics, foundations, blushes, lipsticks, lip balms, eyeliners, mascaras, oil removers, color cosmetic removers, nail polishes, and powders.

### EXAMPLES

- The following examples are presented to further illustrate the compositions and methods of this invention, but are not to be construed as limiting the invention. All parts and percentages in the examples are on a weight basis and all measurements were obtained at 23°C, unless indicated to the contrary.

#### Materials

- The representative (AB)<sub>n</sub> silicone polyethers, herein designated as (AB)<sub>n</sub> SPE, used in the emulsion compositions of the present invention were prepared from the hydrosilylation reaction of *M'D<sub>x</sub>M'* siloxanes (dimethyl-hydrogen terminated (Me<sub>2</sub>HSiO) linear polydimethylsiloxanes of varying degree of polymerizations (as designated by x) and *allyl terminated polyethers* (alpha, omega-diallyloxy polyethers having the average formula (CH<sub>2</sub>=CHCH<sub>2</sub>O(CH<sub>2</sub>CH<sub>2</sub>O)<sub>m</sub>CH<sub>2</sub>CH=CH<sub>2</sub>). Polyglycol AA1200, and AA2000, used as obtained from Clariant (Mt. Holly, NC), contained on average 12 and 22 ethylene oxide units (designated as EO) (i.e. m = 12 and 22).

- (AB)<sub>n</sub> SPE 1A – reaction product from M'D<sub>15</sub>M' and AA2000, M<sub>w</sub> = 16,022  
(AB)<sub>n</sub> SPE 1B – reaction product from M'D<sub>15</sub>M' and AA2000, M<sub>w</sub> = 24,426.  
(AB)<sub>n</sub> SPE 2A – reaction product from M'D<sub>15</sub>M' and AA1200, M<sub>w</sub> = 33,552.  
(AB)<sub>n</sub> SPE 2B – reaction product from M'D<sub>15</sub>M' and AA1200, M<sub>w</sub> = 35,352.

**Example 1***(AB)n SPE 1A dispersion in water*

- 5 (AB)n SPE 1A (reaction product of M'D<sub>15</sub>M' siloxane and polyglycol AA2000 polyether) is a solid, waxy material with a melting point of 45-47 °C. A dispersion of this polymer was made by dispersing this solid polymer in water using a low shear mechanical mixing device. The dispersion has an average particle size of 1.867 µm.

10 **Example 2 - 4***(AB)n SPE 1A dispersions in alcohol-containing water*

- Dispersions of (AB)n SPE 1A copolymer were prepared in alcohol-water mixture. The solid (AB)n SPE copolymer was dispersed, via a mechanical shear device, into isopropanol / water mixtures at a ratio of 5/85, and 20/70, respectively, as summarized in Table 1. As shown in figure 1, smaller dispersion size was obtained when a higher amount of isopropanol was used. Additionally, dispersion of sub-micron size in water was also obtained by vacuum stripping IPA off the mixture to almost alcohol-free.

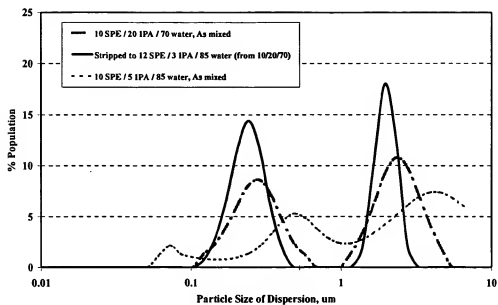
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**Table 1**

<b>Example #</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>
<b>Process Process</b>	<b>As mixed</b>	<b>As mixed</b>	<b>As mixed</b>	<b>Mixed, then stripped</b>
<b>18841-99 (AB)n SPE 1A, g</b>	10.01	30.002	30.02	17.5
<b>IPA, g</b>		15.055	60.314	3.7
<b>D.I. Water, g</b>	90.68	255.106	210.305	122.5
<b>Final Dispersion Composition</b>				
<b>Wt. % Polymer</b>	<b>10</b>	<b>10</b>	<b>10</b>	<b>12</b>
<b>Wt. % IPA</b>	<b>0</b>	<b>5</b>	<b>20</b>	<b>3</b>
<b>Wt. % Water</b>	<b>90</b>	<b>85</b>	<b>70</b>	<b>85</b>
<b>pH of the dispersion</b>	4.89	4.88	5.10	5.17
<b>Avg. dispersion size, µm</b>	<b>1.867</b>	<b>2.102</b>	<b>1.359</b>	<b>0.939</b>
<b>D(v, 0.5), µm</b>	1.316	1.635	1.344	0.303
<b>D(v, 0.9), µm</b>	4.697	4.975	2.959	2.049

- 25 The dispersion particle size profiles of (AB)n SPE in these compositions, SPE/IPA/water @ 10/5/85, 10/20/70, and stripped 12/3/85, are shown in the following graphs.

Figure 1



**Example 5***Vitamin A palmitate loaded (AB)<sub>n</sub> SPE particle dispersions.*

- 5 Vitamin A palmitate is not soluble in water and can not be dispersed in water directly. The example shows that particle dispersion forming (AB)<sub>n</sub> SPE block copolymer can be used to incorporate water-insoluble vitamins and form a stable dispersion in water. The dispersion was prepared as followed: A 50/50 by weight pre-mixture of vitamin A palmitate and 1-2287 vinyl silicone fluid was prepared. The premix was incorporated to form a homogeneous
- 10 mixture with (AB)<sub>n</sub> SPE 1A copolymer. Deionized water was slowly incorporated into the above mixture while under continuous mixing. As shown in Table 2, a dispersion having an average particle size of 1.68  $\mu\text{m}$  was obtained in water. The vitamin A palmitate payload in the dispersion particles was 17%.

15

**Table 2**

Example #	5
Process History	Roto-wheel mixed
18841-99 (AB) <sub>n</sub> SPE 1A, g	7.681
Vitamin A palmitate, g	1.64
1-2287 vinyl fluid, g	1.64
D.I. Water, g	90.5
Total batch, g	101.46
Wt.% (AB) <sub>n</sub> SPE	7.57
Wt.% VAP	1.62
Wt.% Vinyl fluid	1.62
Wt.% Water	89.20
Avg particle size, $\mu\text{m}$	1.676
50%, $\mu\text{m}$	0.8724
90%, $\mu\text{m}$	5.845

**Examples 6 - 8***Si/W emulsions from (AB)<sub>n</sub> SPE*

- 5 Three Si/W emulsions of different compositions were prepared via high shear emulsification process. The method of preparation includes the following steps: the silicone fluids is first incorporated into (AB)<sub>n</sub> SPE 2A copolymer to form a homogeneous mixture. A small amount of water was incorporated into the phase A mixture, followed by high shear mixing to disperse the water using a Speed Mixer. Water additions in small quantity continued until the mixture inverted or form a continuous, smooth cream (called inverted into a water-continuous emulsion concentrate). The remaining water was added to further dilute the emulsion to desired concentration and consistency. The final emulsions have an average particle size between 1.3 and 2.1  $\mu\text{m}$ .
- 10
- 15 These Si/W emulsion examples illustrated that it is possible to prepare emulsion particles of desirable compositions comprising the (AB)<sub>n</sub> SPE polymer and silicone oils. The exact compositions are shown in the Table 3.

**Table 3**

Example #	6	7	8
Phase A			
19056-52 (AB) <sub>n</sub> SPE 2A (15dp + AA1200), g	10	10	20
1-2287 vinyl fluid, g		10	5
DC 245 fluid, g	10		
Phase B			
D.I. Water, g	60	50	61.3
Total batch size, g	80	70	86.3
Wt. % (AB) <sub>n</sub> SPE	12.50	14.29	23.17
Wt. % fluid	12.50	14.29	5.79
Wt. % water	75.00	71.43	71.03
Final appearance:	Smooth, milky white emulsion	Smooth, milky creamy dispersion	Smooth, thick, milky white emulsion
Avg. particle size, $\mu\text{m}$	2.028	1.312	1.617
D(v, 0.5), $\mu\text{m}$	1.281	1.044	1.809
D(V, 0.9), $\mu\text{m}$	5.06	1.925	2.851

**Examples 9 –10***Sub-micron (AB)<sub>n</sub> SPE 1B copolymer particle emulsions in water*

- 5 Another (AB)<sub>n</sub> SPE block copolymer was used to prepare Si/W emulsions. (AB)<sub>n</sub> SPE 1B is the block copolymer reaction product of M'D<sub>15</sub>M' siloxane and polyglycol AA2000 polyether (segment length of 22 EO units) and has a melting temperature 45 – 47 °C.
- 10 The Si/W emulsions for Examples 9 and 10 were made by mechanical emulsification using a high-shear mixer (Speed Mixer), similar to the ones described above. The step-wise procedures can be found in the previous examples. The final Si/W emulsions had an average particle size of 0.394 μm and 0.725 μm, respectively, as summarized in Table 4 below.

**Table 4**

Example#	9	10
Phase A		
(AB) <sub>n</sub> SPE 1B, g	10	10
1-2287 vinyl fluid, g	10	
DC 245 fluid, g		10
Phase B		
D.I. Water, g	50.3	60
Total batch size, g	70.3	80
Wt. % SPE	14.22	12.50
Wt.% fluid	14.22	12.50
Wt.% water	71.55	75.00
Final appearance:	Smooth, thin milky dispersion	Cloudy dispersion with slight creamy feel
Avg. particle size, μm	0.394	0.725
D(v, 0.5), μm	0.296	0.619
D(V, 0.9), μm	0.815	1.405

**Examples 11 - 13***Si/W emulsions and vitamin loaded (Si+O)/W emulsions*

- 5 (AB)<sub>n</sub> SPE block copolymer in Si/W emulsion form can be used to carry and protect water-insoluble oils and substances. These emulsions can subsequently be incorporated into water-based end products and formulations.
- 10 Vitamin A palmitate is water-insoluble and cannot be incorporated directly into water-based formulations. These examples showed that stable Si/W emulsions containing various amount of vitamin A palmitate were successfully prepared from (AB)<sub>n</sub> SPE block copolymer.
- 15 The Si/W emulsions were prepared using SPE 2A copolymer, an (AB)<sub>n</sub> block copolymer product of M'D<sub>15</sub>M' siloxane and polyglycol AA1200 polyether and has a melting temperature of 27 – 32 °C. DC 245 silicone cyclics and 1-2287 vinyl silicone fluids were used to prepare the following Si/W and (Si+O)/W emulsions.
- 20 These emulsions were prepared following the following procedures: vitamin A palmitate was first mixed with 1-2287 vinyl silicone fluid to form a homogeneous mixture, then incorporated into the (AB)<sub>n</sub> SPE 2A copolymer with mixing to yield a homogeneous premixture. De-ionized water was slowly and gradually incorporated into the phase A mixture using a high-shear mixer (Speed Mixer) till the mixture inverted into water-continuous mixture. The remaining water was added, under shear to complete the dilution to
- 25 desired composition. The final emulsions are smooth, milky white emulsions, as summarized in Table 5.
- 30 The two vitamin A palmitate loaded (AB)<sub>n</sub> SPE block copolymer emulsions had a particle size of 1.62 μm and 1.02 μm, respectively. The vitamin payload was 13.4 % and 20.3%, respectively.



Table 5

ID example	11	12	13
<b>Phase A</b>			
(AB)n SPE 2A, g	20	20	20
1-2287 vinyl fluid, g	5	5.1	3.1
Vitamin A palmitate, g		3.1	5.1
<b>Phase B</b>			
D.I. water, g	61.3	60.8	60.9
Batch size, g	86.3	89	89.1
Wt. % SPE	23.17	22.47	22.45
Wt. % VAP	0.00	3.48	5.72
Wt.% fluid	5.79	5.73	3.48
Wt.% water	71.03	68.31	68.35
Final Dispersion Appearance	Smooth, white creamy emulsion	Smooth, beige emulsion with some viscosity	Smooth, light yellowish emulsion with some viscosity
Avg. particle size, $\mu\text{m}$	0.369	1.617	1.021
D(v, 0.5), $\mu\text{m}$	0.358	1.809	0.862
D(v, 0.9), $\mu\text{m}$	0.539	2.851	2.046

5

The particle size profiles of these Si/W and (Si+O)/W emulsions are shown in figure 2.

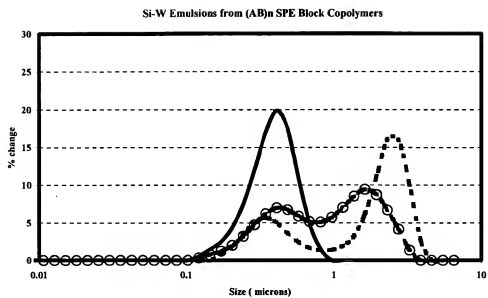
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Figure 2



**Examples 14 - 15***Vitamin loaded emulsions formulated into skin care products*

- 5 Vitamin A palmitate loaded (AB)<sub>n</sub> SPE particle dispersions in water were prepared according to the method shown in the previous examples. Example 14 was prepared from (AB)<sub>n</sub> SPE copolymer of 15 dp siloxane and Polyglycol AA2000 polyether, and Example 15 dispersion from (AB)<sub>n</sub> SPE copolymer of 15 dp siloxane and Polyglycol AA1200 polyether. The final compositions of these dispersions are shown in Table 6.

10

**Table 6**

Example #	14	15
SPE Polymer type	(AB) <sub>n</sub> SPE 1B	(AB) <sub>n</sub> SPE 2B
Main SPE structure	15 dp siloxane & AA2000 polyether	15 dp siloxane & AA1200 polyether
% SPE Polymer	21.12	21.17
Wt. % VAP	4.23	4.24
% Tocopherol	0.42	0.46
% 1-2287 Silicone fluid	4.23	4.25
% Water	70.00	69.89
Appearance of dispersion	Light yellowish, creamy thick paste	Light yellowish, creamy paste
PH of dispersion	5.29	3.58
Avg. particle size, $\mu\text{m}$	0.326	0.355

15

The vitamin-loaded SPE particle dispersions were formulated into skin care formulations.

**Oil-in-water body lotion**

<b>Ingredients</b>	<b>Parts</b>
--------------------	--------------

**Part A**

Cetearyl Alcohol	3
Diisopropyl Adipate (Crodamol DA)	5
Dimethicone (Dow Corning Silicone 200 / 100 cs)	0.5
Potassium cetyl phosphate	1.5
Buthylated hydroxytoluene	0.05
Cheating agent (EDTA)	0.1
Phenoxyethanol	0.6

**Part B**

Water	up to 100
Carbomer 980 thickener	30
Potassium hydroxide	1.5

**Part C**

Vitamin A palmitate loaded SPE particle dispersions	19.88
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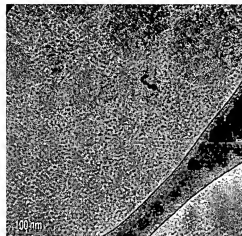
- 5 To prepare the body lotion, the following procedure was followed: The ingredients in Part A were mixed and heated to 85 °C to homogeneous. Cool the part A mixture to 40 °C, then incorporate the part B ingredients. Cooled the mixture to ambient temperature. Incorporate vitamin A palmitate-loaded SPE particle dispersion into the mixture and mix to homogeneous. The final mixture is a smooth, slightly yellowish creamy lotion.

- 10 A cryo-TEM image of the “as prepared” body lotion illustrated in Example A, prepared from (AB)<sub>n</sub> SPE particle dispersion was shown below. The characteristic particle structures uniquely associated with the (AB)<sub>n</sub> SPE dispersion particles are shown in figure 3.

Figure 3

5

Cryo-TEM image of body lotion formulated from vitamin-loaded SPE particle dispersions



### Moisturing Gel for Skin

- 5 The (AB)<sub>n</sub> type SPE particle dispersions can be formulated into aqueous based gel formulations. SPE vesicles provide a convenient means to incorporate oil-soluble vitamins into water-rich gel formulations.

**Ingredients****Parts****Part A**

Water	to 100%
Preservative	0.30%
Polyacrylamide, C13-14 Isoparaffin, laureth-7 (Sepigel 305)	1%

**Part B**

Vitamin A palmitate-loaded SPE particle dispersions	19.88
--	-------

- 10 To prepare the gel, the following procedure was followed: The ingredients in Part A were mixed to homogeneous. Vitamin A palmitate-loaded SPE particle dispersion was then incorporated and mixed to homogeneous. The final product is a beige, smooth gel.

- 15 To further demonstrate the integrity of the dispersion particles in formulations, cryo-TEM images of the "as formulated" products were taken. An image of the gel from the above example is shown below in figure 4. As illustrated, the dispersion particles were well preserved.

20

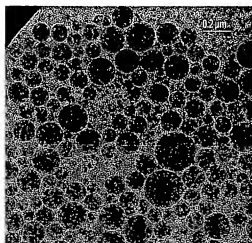
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Figure 4.

Cryo-TEM image of body lotion formulated  
from vitamin-loaded SPE particle dispersions



**Examples 16-17**

The following vitamin A palmitate loaded (AB)<sub>n</sub> SPE particle dispersions in water were prepared according to the method shown in the previous examples of this invention. An

- 5 The emulsion labeled as Example 16 was prepared from (AB)<sub>n</sub> SPE 2A, a copolymer of 15 dp siloxane and Polyglycol AA2000 polyether, and the emulsion labeled as Example 17 from (AB)<sub>n</sub> SPE 1B, a copolymer of 15 dp siloxane and Polyglycol AA2000 polyether. The final compositions of these dispersions are shown in the following table. In this case, no water-miscible solvent was required. DC 1-2287, a methylvinylsilicone cyclics (from Dow Corning Corp.) was used. The compositions of these two emulsions are shown in Table 5 below.

The stability of the emulsions prepared from (AB)<sub>n</sub> SPE copolymers are also shown. The particle sizes of these emulsions after 5 weeks aging @ 40°C were found comparable to their initial values, as illustrated in the table.

15

**Table 7**

Example #	16	17
(AB) <sub>n</sub> SPE type	(AB) <sub>n</sub> SPE 2A	(AB) <sub>n</sub> SPE 1B
Carrier Fluid	1-2287	1-2287
Wt. % (AB) <sub>n</sub> SPE	20.13	20.31
Wt. % VAP	4.15	4.10
Wt.% silicone fluid	4.14	4.44
Wt.% water	71.58	71.15
Dispersion appearance	Yellow, creamy, uniform emulsion	Yellow uniform emulsion; moderate viscosity
Initial particle size, um	0.973	0.242
Particle size after 5 weeks @ 40 C, um	0.965	0.398

For purpose of further showing the uniform quality of emulsion particles formed from (AB)<sub>n</sub> silicone polyether copolymers, an optical micrograph of the Example 16 emulsion is shown below.

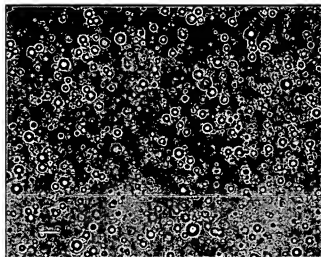
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Figure 5

Optical micrograph of Example 16 emulsion at 1000X magnification (bar represents 5  $\mu\text{m}$ )

5



## CLAIMS

1. A water continuous emulsion composition comprising a  $(AB)_n$  silicone polyether  
5 copolymer wherein the emulsion has an average particle size of less 10 micrometers.
2. The emulsion composition of claim 1 wherein the  $(AB)_n$  silicone polyether copolymer is a polyorganosiloxane-polyoxyalkylene block copolymer having the average formula;
- 10 
$$-[R^1(R_2SiO)_x(R_2SiR^1O)(C_mH_{2m}O)_y]_z-$$
  
where  
x and y are greater than 4, m is from 2 to 4 inclusive, z is greater than 2,  
R is independently a monovalent organic group,  
R<sup>1</sup> is a divalent hydrocarbon containing 2 to 30 carbons.
- 15 3. The emulsion composition of claim 2 wherein x ranges from 5 to 19, and m is 2.
4. The emulsion composition of claim 1 further comprising a water miscible volatile solvent.
- 20 5. The emulsion composition of claim 4 where the solvent is an alcohol.
6. The emulsion composition of claim 1 further comprising an organopolysiloxane.
7. The emulsion composition of claim 6 where the organopolysiloxane is a volatile siloxane.
- 25 8. The emulsion composition of claim 7 wherein the organopolysiloxane is a vinyl functional organopolysiloxane.
9. The emulsion composition of any one of claims 1 – 8 further comprising a personal,  
30 household, or healthcare active ingredient.

10. A personal, household, or healthcare composition comprising the emulsion composition of any one of claims 1 – 9.
11. A process for preparing a water continuous emulsion having an average particle size of less than 10 micrometers comprising;
- 5 I) mixing
- A)  $(AB)_n$  silicone polyether copolymer
- B) a water miscible volatile solvent
- to form a hydrophobic phase,
- 10 II) adding water to the hydrophobic phase to form the water continuous emulsion.
12. The process of claim 11 wherein C) a silicone or organic oil is included in the mixing of step I).
13. The process of claim 12 wherein the silicone is a volatile methyl siloxane.
14. The process of claim 12 wherein the silicone is a vinyl functional organopolysiloxane.
15. The process of claim 11 or 12 wherein step I further comprises;
- 20 D) a personal, household, or healthcare active.
16. The product produced by any one of claims 1 to 15.

**ABSTRACT**

Emulsion compositions from  $(AB)_n$  silicone polyether block copolymers, methods for preparing the emulsion compositions, and personal, household, and healthcare formulations  
5 containing the emulsion compositions are disclosed.